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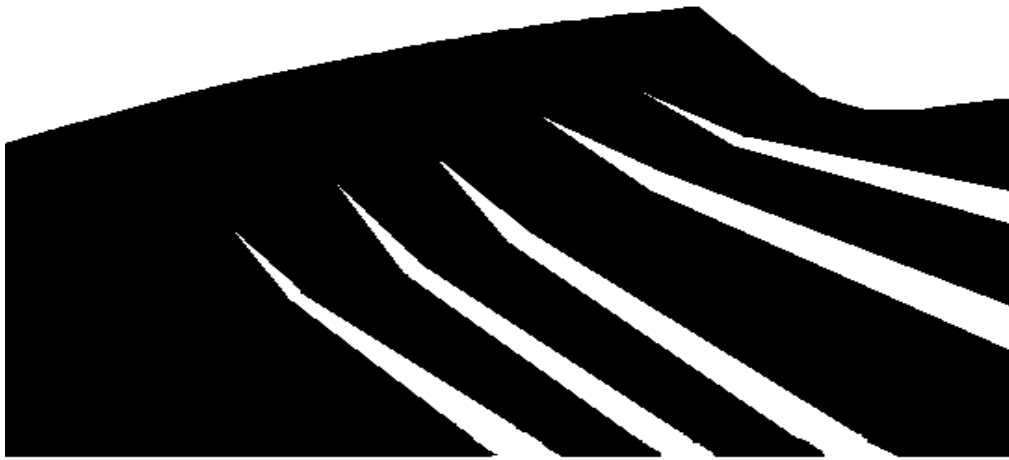
December 23, 1996

LANL-CST-DP-63, R5

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PREPARATION OF CORE SAMPLES FOR CRUSHED ROCK EXPERIMENTS

LOS ALAMOS ***QUALITY PROGRAM***



APPROVAL FOR RELEASE

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Signature on file

DATE

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Los Alamos

Yucca Mountain Site

Characterization Project

HISTORY OF REVISION

REVISION NO.	EFFECTIVE DATE	PAGES REVISED	REASON FOR CHANGE
R0	03/20/87	N/A	Initial procedure.
R1	03/27/89	N/A	History of revisions not required until Rev. 3.
R2	10/21/91	N/A	History of revisions not required until Rev. 3.
R3	08/13/92	All	Complete rewrite. This procedure was formally identified as LANL-INC-DP-63.
R4	07/08/94	All	Complete rewrite. Update to new format of QP-06.3.
R5	12/23/96	All	Revised to comply with LANL-YMP-QP-06.3 requirements.

Los AlamosYucca Mountain Site
Characterization Project

PREPARATION OF CORE SAMPLES FOR CRUSHED ROCK EXPERIMENTS

1.0 PURPOSE

The purpose of this detailed technical procedure (DP) is to describe the grinding, sieving, and washing of YMP core samples and pure mineral specimens, and to obtain representative fractions of these samples for performing multiple tests. Experiments can be performed utilizing sample subsets which are representative of the original sample. These experiments are conducted at Los Alamos National Laboratory (LANL) for the Yucca Mountain Site Characterization Project (YMP).

2.0 SCOPE

This DP applies to the preparation and representative sampling of YMP samples and minerals utilized in the Batch Sorption and Dynamic Transport Studies. The representative sampling of these crushed geologic samples are in the 75-500 micron range for sorption and crushed rock column experiments and other analyses.

3.0 REFERENCES

LANL-YMP-QP-02.7, Personnel Training
LANL-YMP-QP-03.5, Documenting Scientific Investigations
LANL-YMP-QP-08.1, Identification and Control of Samples
LANL-YMP-QP-12.3, Control of Measuring and Test Equipment and Standards
LANL-YMP-QP-17.6, Records Management
LANL-CST-DP-99, Collection of Bulk Well and Spring Water Samples
CST Division ES&H Operational Policy

4.0 DEFINITIONS

4.1 Sample

Specimens of materials intended for laboratory studies or analyses.

4.2 Representative Sample

A portion of material whose composition and representative of the original sample.

5.0 RESPONSIBILITIES

The following personnel are responsible for the activities identified in Section 6.0 of this procedure.

- Principal Investigator (PI)
- Users

6.0 PROCEDURE

The use of this procedure must be controlled as follows:

- If this procedure cannot be implemented as written, YMP personnel should notify appropriate supervision. If it is determined that a portion of the work cannot be accomplished as described in this DP, or would result in an undesirable situation, that portion of the work will be stopped and not resumed until this procedure is modified, replaced by a new document, or the current work practice is documented in accordance with QP-03.5, Section 6.1.6..
- Employees may use copies of this procedure printed from the controlled document electronic file; however, employees are responsible for assuring that the correct revision of this procedure is used.
- When this procedure becomes obsolete or superseded, it must be destroyed or marked “superseded” to ensure that this document is not used to perform work.

6.1 Principle

This procedure describes the preparation of crushed material which will be used in sorption and crushed rock column experiments to determine the sorption of radionuclides. The material is crushed to speed up the reaction times and provide a more efficient and easily modeled geometry for the sorption. Sample particle size and surface interactions are the subject of sorption study tests. Reproducibility of these tests requires that each fraction be representative of the original sample. The rotary sample dividing technique is an accepted method for ensuring each fraction has similar particle distribution and properties of the original sample.

6.2 Equipment and Hardware/Software

Equipment needed to prepare the core are listed below. Items equivalent to those listed below may be used provided they perform the same function with an acceptable level of performance as judged by the PI.

- jaw crusher
- agate grinding set, Fritsch or equivalent
- ASTM sieves with a catch pan on bottom
- equipment of non-ferrous material such as glass or teflon beakers, plastic bottles, and teflon coated spatulas

- balance with a capacity of 1500 g and a tolerance of .01 g
- stainless steel hammer and stainless steel crushing block

6.2.1 Equipment Malfunctions

There are no critical equipment malfunctions that are likely to occur that would interrupt this procedure.

6.2.2 Safety Considerations

Ensure compliance with CST Division Environmental Safety and Health Operational Policy.

6.2.3 Special Handling

None required unless specified by the PI.

6.3 Preparatory Verification

6.3.1 Hold Points

There are no hold points for this procedure.

6.3.2 Calibration

Balances used for weighing are required to be calibrated pursuant to LANL-YMP-QP-12.3. When data are collected from a balance, the unique identifier number of that balance is recorded in the user's laboratory notebook or binder along with the data collected. Proper procedure for documenting this data is provided in LANL-YMP-QP-03.5.

ASTM sieves are not calibrated and are replaced when damage is evident.

6.3.3 Environmental Conditions

No special environmental conditions are required for this DP. If any special conditions are used, they will be recorded according to Section 6.7 of this DP.

6.4 Control of Samples

Sample code numbers are referenced in the YMP Solid Binder (a YMP controlled notebook). All samples will be controlled according to LANL-YMP-QP-08.1, Identification and Control of Solid Samples.

6.5 Implementing Procedure

6.5.1 Preparation of Crushed Sample

- 6.5.1.1 Obtain a portion of tuff or pure mineral specimen from the cabinets in the basement of RC-1 at TA-48.
- 6.5.1.2 Break YMP core or tuff sample into small pieces using a stainless steel hammer. Save a piece (about 25 g) for alter mineralogic analysis (x-ray and thin section analyses).
- 6.5.1.3 Crush the core into a coarse material (gravel sized). Alternatively, the core can be crushed utilizing a stainless steel hammer and stainless steel crushing block.
- 6.5.1.4 Add a small amount of the material to the agate grinding vessel or jaw crusher. Grind the material until a fine powder is made.
- 6.5.1.5 Place the fine powder into ASTM sieves (sizes determined by the PI and recorded in laboratory notebook) and pass as much of the material as possible through the sieves.
- 6.5.1.6 Regrind any of the material that will not pass through the top sieve and resieve. Repeat this step until all material is processed; this assures a more representative mixture.
- 6.5.1.7 Enter necessary data in laboratory notebook (See Attachment 1)

- 6.5.2 If the PI determines the ground material is to be washed, one of the following methods should be utilized. If natural water is used to wash the ground material, LANL-CST-DP-99 must be followed to obtain the water.

6.5.2.1 Method #1

- a) Place a portion of dry sieved material (Section 6.5.1) into a large beaker and fill the beaker with the water specified by the PI. Mix thoroughly and let the material settle. Pour off the cloudy water into the sink drain.
- b) Repeat the water wash until the water remains clear after settling (more than 20 washes is not unusual).
- c) Cover the beaker and let the wet sieved material air dry or place into a vacuum oven with low heat (no more than 30°C) and apply house vacuum.
- d) Save a 10 g portion of the washed material for mineralogic and chemical analysis.

- e) Place the remaining material into suitable container.
- f) Label the container with sample ID, weight, date and initials. Enter this information in laboratory notebook (See Attachment 2).

6.5.2.2 Method #2

- a) Transfer the desired portion of the dry sieved material to the lower boundary sieve.
- b) Use a portion of the water specified by the PI for washing the dry sieved material.
- c) If a large amount of material is to be washed, the sieve may be partially submerged in a tray of the water and stirred to dislodge small particles.
- d) Pass the water through the sieve containing the dry sieved material. Continue until particles no longer pass through the sieve.
- e) Place a cover on the sieve containing the washed material and set aside to dry or place into a vacuum oven with low heat (no more than 30°C) and apply house vacuum.
- f) Save a 10 g portion of the washed material for mineralogic analysis and chemical analysis.
- g) Place the remaining wet sieved crushed material into a suitable container.
- h) Label the container with sample ID, weight, date and initials. Enter this information in laboratory notebook (See Attachment 2).

6.5.3 Representative Sampling With Endecotts Rotary Sampling Divider

- 6.5.3.1 For batch experiments and crushed rock columns, the particle size of the dried minerals should range from 75-500 microns. Weigh out a sufficient amount in a weighing boat. Record weight and unique identifier number of balance. Before adding the contents of the boat to the Endecotts Rotary Sample Divider, be sure the Rotary divider has been cleaned with distilled water and dried.
- 6.5.3.2 Close the valve in the Hopper turning counter clockwise. Place all 10 sample bottles in the correct position.
- 6.5.3.3 Add the weighed sample and turn on the main switch to the "I" position and check that the main indicator light is on.
- 6.5.3.4 Slowly open the Hopper valve by turning clockwise.

- 6.5.3.5 The minimum time for all samples to flow to the containers should be greater than 15 seconds for representative sampling. If less than 15 seconds, repeat the process.
 - 6.5.3.6 After dividing samples, take the containers out for weighing, labeling, and dating. Record weight and unique identifier number of balance.
 - 6.5.3.7 Take apart the Rotary Sample Divider for cleaning. The Hopper and Valve Assembly are usually cleaned by rinsing with deionized water three times and dried without permitting contamination. Deviation from this cleaning routine will be documented in Section 6.7 of this procedure.
- 6.5.4 Ensure that the following entries have been recorded in the laboratory binder:
- Unique identifier of the sample.
 - Date grinding of sample was completed.
 - Sizes of sieves used.
 - Amount of dry sieved material. Location of ground material.
 - Date of sample was washed.
 - Washing method used.
 - Source of water used for washing.
 - Amount washed.
 - Amount recovered after washing and drying.
 - Location of ground and washed material.
 - Name of individual(s) performing the grinding and washing
 - Unique identification of balance.

6.6 Data Acquisition and Reduction

The active recording of data as specified in Section 6.5.4 will constitute the data acquisition. Computer programs such as word processing editors and spreadsheets will be used for recording and formatting data but are not part of the data acceptance criteria. No data reduction is employed in this DP.

6.7 Deviations from the DP and Potential Sources of Error and Uncertainty

Deviations from this procedure shall be documented in the user's notebook and approved by the PI (Initial/Sign, Date). If the deviations are deemed critical by

the PI, documentation shall include an evaluation addressing the impact of potential sources of error and uncertainty.

7.0 RECORDS

Records generated as a result of this DP are entries in laboratory notebooks or attachments to laboratory notebooks. The documentation should consist of any applicable items identified in Section 6.0 of this procedure. Laboratory notebooks should be kept in accordance with QP-03.5.

All records should be submitted to the Records Processing Center in accordance with QP-17.6.

8.0 ACCEPTANCE CRITERIA

Proper recording of the data specified in Section 6.5.4 constitute the acceptance criteria for this DP. If no critical deviations (see Section 6.7) were made, these data will be accepted as qualified data for YMP.

9.0 TRAINING

- 9.1 Prior to conducting work described in Section 6.0, the user requires training to this procedure.
- 9.2 Training to this procedure is accomplished by “read only”. Training will be documented in accordance with QP-02.7.

10.0 ATTACHMENTS

The forms shown are suggested formats. Other formats may be used but must contain, as a minimum, the information contained on Attachments 1 and 2.

Attachment 1: Sample Grinding Using LANL-CST-DP-63

Attachment 2: Sample Washing Using LANL-CST-DP-63

SAMPLE GRINDING USING LANL-CST-DP-63

SAMPLE ID CODE:

Purpose:

Date Grinding of Sample was completed:

Amount of Dry-Sieved Material ($>75 \mu m$):

Amount of Dry-Sieved Material (75 - 500 μm):

Amount of Dry-Sieved Material ($>500 \mu m$):

Location of Ground Material:

Individual Performing Grinding:

Unique Identifier of Balance:

EXAMPLE

Data Entered By:

6/9/94

Date Verified By:

6/9/94

SAMPLE WASHING USING LANL-CST-DP-63

SAMPLE ID CODE:

Purpose:

Date Sample was Washed:

Washing Method Used:

Source of Water Used for Washing:

Size of Material Washed: 75-500 μm

Method Used for Drying: Vacuum Oven at 102 ° F

% Recovered after Washing and Drying _____ %

Location of Ground and Washed Material: TA-48, PC-1, Room _____

Individual Performing Washing:

Amount washed in grams: _____ grams

Amount recovered after washing and drying: _____ grams

Unique Identifier of Balance:

Data Entered by:

Date: June 9, 1994

Data Verified by:

Date: June 9, 1994